

CEUR # 0519107 Rev 100 9 J 27-2

Alkyne Preparation by HEDR-HDI

from FURACA # 06 by 1st method

RAW MATERIALS

FURACA #06 (wet) 116.8g (9.10 50.09)

Dmso 443.2 g ml

THE 500.0 g

MAEM 74.0g

TEA 40.0 ml

ELOAC (750 + 400) ml (10)

Dmso 200.0 ml

Nacl 135.0 g

THE (950 + 100) ml

carbon 15.0g

cor. HCl (21.0 + 19.5 + 12.0) ml

IPE 450.0 ml

IPE (washing) 250.0 ml

EDTA 1.0g

[500 - (wet wt - 60)]

PROCEDURE

1. charge Dmso & THF then cool to 5°C

2. charge wet FURACA followed by MAEM  
at 3-5°C

3. Add TEA in 3 hrs at 3-5°C

con't maintain the temp at 3-5°C

upto complete run

4. charge ELOAC 750 ml <sup>and EDTA</sup> & stir at 15°C for 15  
hrs! separate the layers

5. Extract the ag. layer = 200 ml <sup>ECO A</sup>  
for 15' & separate the layers.  
(settle it for 15')

6. Extract the mixed stock layer  
(20.0g = 1st trc)

7. 200 ml DMW and Irgan  
 add this aq. soln into rich org. phase  
 meth. 7. To this aq. soln add NaCl &  
 950 ml THF at 18-20°C  
 8. Adjust the pH to 3.0-3.1. to corr. HCl  
 at 18-20°C in 20-25'  
 9. Separate the layers & eliminate  
 the org. phase  
 10. charcolise the org. phase 23°F for 40'  
 at 18-20°C  
 11. filter it & wash the bed to 100ml THF  
 12. Adjust the pH to 1.0-0.9 by  
 corr. HCl. in 10-15' at 18-20°C  
 13. seed to 1.0g CFUR HCl and  
 stir for 1hr at 18-20°C  
 14. Again bring the pH to 1.0-0.9  
 by corr. HCl at 18-20°C  
 15. seed again to 1.0g CFUR HCl &  
 stir for 1hr at 18-20°C  
 16. Add 450 ml IPE in 40-45' at 18-20°C  
 17. stir for 1hr at 18-20°C & filter it.  
 18. wash the product to 250 ml IPE  
 19. Dry the product over at 40°C up to w/w %  
 MC < 5.0%

DURATION RUN MONITORING RESULTS

	FURACA	CFUR	MAEM	CFT	MBT
18hr.	2.43	76.33	5.97	0.38	12.74

Terminated in 18<sup>1</sup> hr  
18hr.

FLO  
05/96

OBSERVATION:

1. Reaction is main in clear sol? after 10 hr. 5 min.  
but very dark in colour
2. Vol. of Ag. sol? is 940.0 ml
3. Vol. of EtOAc layer is 1570.0 ml
4. pH of Ag. sol? + NaCl + THF is 8.30
5. Vol. of CFUR Rich THF layer is 1135.0 ml (B.C)
6. Vol. of Ag. sol? containing NaCl is 790.0 ml
7. After 30% charactherization, colour of the rich to sol? becomes lighter like STD.
8. Only 0.5g CFUR HCl is needed, in 1hr stirring about amount of material comes out.
9. Colour of slurry is better than #03 & 04.
10. Colour of dried product is slightly better known #03 & 04.

Dry wt	CFUR Assay : CFT	Imp. Formic m/c
87.4 g	97.45	4.3%
	97.45 0.59	0.37 ± 0.18% qualitative

CV: 0.27